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MULTI WALL CARBON NANOTUBE REINFORCED SILICONE FOR AEROSPACE APPLICATIONS

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Original Article

ABSTRACT

Product design firstly requires the clear understanding of the requirement of the product. Strength and cost of the material are predominantly considered during the design. The strength of any material can be increased by incorporating fillers of the requirement. The percentage of filler, adhesion of filler with the material matrix, processing method is some parameters deciding the mechanical properties of the resultant composite. This research is focused on increasing the mechanical properties of Silicone elastomer by loading of 5wt% of multi-wall carbon nanotubes (MWCNTs) as fillers. The Silicone/MWCNT composite was masticated in two roll milling followed by compression molding. The tensile properties were measured through experiments as per ASTM D412 standard. The tensile strength, modulus and elongation at break of the filled nanocomposite are 7.21 MPa, 1.673 MPa and 235 mm. The compression strength of the filled composite was observed as 18.6 MPa at 83.3% of the compression. The shore hardness of the filled composite was increased from 40 to 64. It is perceived that the mechanical property of the filled nanocomposite is drastically increased by loading of MWCNTs. Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM) studies were also done to understand the dispersion of the compound and mechanical damage in the developed composite. The results have shown the good wetting and high diffusion of nanofillers that led to the strong interfacial synergy interaction with silicone matrix. The resultant nanocomposite is regarded to be a high strength elastomeric material for aerospace product design.

KEYWORDS**: *Multi-Wall Carbon Nanotubes, Silicone Elastomer, Nanofillers & Compression Molding***

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INTRODUCTION

The materials with improved mechanical properties can be achieved by focusing on filler material, filler size and aspect ratio. The Inclusion of a particular percentage of nanofillers may depict enhanced mechanical properties on resultant composites [1]. In recent years, the carbon nanotube has received a special attention among researchers as a filler material due to its unique properties like high modulus and tensile strength [2]. Solution casting, melt-mixing, and in situ polymerization methods are preferred for the preparation of nanocomposites, where fillers are added with rubber matrix to incur required properties. Silicone has been used in numerous medical and aerospace applications due to its low thermal conductivity, low chemical reactivity, low toxic, good thermal stability and high gas permeability. They are available in different grades and different

forms like gel, liquid. The room temperature vulcanizes (RTV) silicone is in the form of liquid [1]. A medical grade of silicone is used in medical applications and transplantations. The mechanical strength of silicone is noticed to be a concern in the product design. The synthe size of a silicone composite with high strength will be very useful towards the development of many products. The required configuration or outcome of thermoset polymers is obtained through cross-linking polymerization process [1, 3-4].

In the past, carbon nanotubes were attempted for the reinforcement of natural rubber, epoxy, polypropylene etc. Bokobza [5] studied the response of 1, 2, 3, 5, 10 PHR (parts per unit weight of rubber) of MWCNT reinforcement on the mechanical and electrical properties of natural rubber (NR). The Young's modulus and tensile strength of the samples are considerably lower than unfilled samples. Elango et al [6] studied the reinforcement of 1, 5, 10, 20, 30 PHR of MWCNT in NR and revealed that mechanical properties are improved as filler increases. Also reported that ductility of the NR decreases as the filler increases and particularly not advisable to use more than 30 PHR of filler for the reinforcement. Bokobza used solution mixing technique and Elango et al used compression molding for the synthesis of the nanocomposite.

Rattanasom [7] attempted silica/carbon black as hybrid filler for the reinforcement of NR and reported that the hybrid vulcanizates containing 20-30 phr of silica as a filler exhibited the enhanced mechanical properties and abrasion, crack growth, rolling, heat growth resistance. Sadhana Agrawal [8-9] reported that 2 PHR MWCNTs filled nanocomposites of silicone rubber fabricated by two roll mixing mill with various concentration of blowing agents has a high range of electrical conductivity and mechanical properties. The 1, 3, 5, 7 wt% of multiwall carbon nanotube reinforced natural rubber composites were fabricated through melt blending method by Tarawneh et al [10] and reported that 5 and 7% of MWCNTs shows better mechanical strength than 1 and 3% reinforcements [10].

In [11], the aspect ratio, specific surface area (SSA) and amino-function of epoxy matrix composites with respect to various filler reinforcements like single-wall carbon nanotubes, double-wall carbon nanotubes, multi-wall carbon nanotubes and carbon blacks were investigated by Florian H. Gojny and Wichmann. They prepared specimens by shear mixing and room temperature cooling method and stated that CNT’s reinforced resin system showed enhanced tensile properties and better dispersion.

Wei Chen [12] achieved higher modulus and tensile strength from polyurethane (PU) composite fiber reinforced with 9.3 wt% of Multi-wall carbon nanotubes. Results pointed out that the diffusion of MWCNTs in polyurethane matrix and interfacial bonding between oxidized MWCNTs and PU matrix are the main reasons for higher mechanical properties of resultant composites. The relationship between mechanical properties and processing conditions of multi walled nanotubes fabricated by spin casting process were investigated by Safadi [13-14]. He stated that 2.5 vol% of MWCNTs inclusion with polymer composites doubled its modulus of elasticity. The inclusion of a small amount of multi walled carbon nanotubes in styrene-b-butadiene-b-styrene (SBS) matrix significantly improved creep resistance, thermal and mechanical properties [15]. The proportion and rate of dispersion of filler material in the polymer composites is a major factor for obtaining higher mechanical and electrical properties. Polymer composite films with brittle, ductile nature can be produced by high and low reinforcement content of nanotubes respectively [6, 16-20].

The intention of the proposed research is to synthesis silicone/5% MWCNT nanocomposite and investigate the effect of filler in regard to mechanical properties.

SYNTHESIS AND MECHANICAL CHARACTERIZATION

MWCNTs (NC7000™ series) were purchased from NANOCYL®, Belgium. They are thin, tube shaped, black in color, fabricated via the Catalytic Chemical Vapor Deposition (CCVD) process. They are significantly made of carbon atoms with a nanometric diameter (9 nm) and micrometer in length (150 µm). Figure 1 shows the TEM image of the MWCNT filler as received from the supplier. Table 1 depicts the properties of the polymer base matrix and filler.

Table 1 Properties of Polymer Base Matrix

Description	Hardness (Shore A)	Density (g/cm^3)	Elongation at Break	Tensile Strength (MPa)
Pure Silicone (L=1.5 µm, d = 9.5 nm, ρ = 1.3 g/cc)	40	1.39	320 %	5.7

The polymer base matrix is Silicone 40, which is mostly used in aerospace applications for preparing the small parts like a washer, rings etc. The predetermined quantity of gel form of silicone 40 was firstly masticated in two roll mill with Luperox 101, an organic peroxide, type D liquid (Hazard Code 5.2; UN 3105; PG II) for about 20 minutes. Luprox 101 was used as a cross-linker that decomposes the silicone matrix and forms high reactive radicals which link the polymer chains chemically and results from a highly elastic and three-dimensional network at the elevated temperatures. The roller gap of 1 mm was maintained and the masticated sheets were rolled up, folded back into the center of the nip to speed up and optimize the homogenization.

The roller gap was adjusted to 0.1 mm after 20 minutes and the predetermined quantity of MWCNTs was slowly added to the mixture. The mixing of the silicone matrix and filler was continued further for 20 minutes in order to have high wetting and interfacial bonding. After ensuring the homogeneous mixing, the milled mixture was then placed into the mold of size 150 mm x 100 mm x 2 mm to prepare samples for tensile testing. The vulcanization was done in the compression-molding machine for about 20 minutes at 160°C to prepare composites in the form of sheet samples. The composite was then post-cured for about 4-5 hours at 210°C in a digital hot air oven. The tensile (dumbbell) samples of ASTM D412 (ISO 37) C-Type standard were prepared from the vulcanized sheet. Meanwhile, the masticated polymer nanocomposite was placed into the mold of 12 mm x 27 mm and the same vulcanization process was followed to prepare cylindrical samples for the compression test.

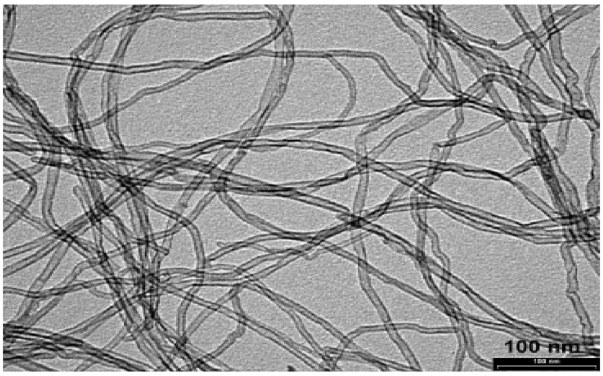

Figure 1: NC7000™ MWCNT – Scale: 100 nm – TEM (as Received from the Supplier)

Figure 2: Specimens used in Tensile Test and Compression Test

Instron® 5582 universal testing machine equipped with standard video extensometer and pneumatic gripper was used for tensile tests. Two equidistant marks in the distance of 33 mm were done on the reduced section from the center of the dumbbell sample to measure the elongation. The double sided tape was put at both ends of the sample to have more frictional grip during the testing. Firstly, Silicone/5 PHR MWCNT sample was attached to the pneumatic gripper and load was gradually applied at a low strain rate of 250 mm/minute till it fractures. The strain-stress data, elongation, linear modulus and tensile stress were recorded. The test was repeated for four times and the mean value was computed. Secondly, the cylindrical sample of 12 mm diameter was placed on the table of uniaxial tensile testing machine and the load was gradually applied at the strain rate of 12 mm/minute to 83.3% of the compression (10 mm). The respective compression load, deformation and compression stress were measured during a testing of each sample and mean of the measurements was recorded. Figure 2 shows the specimens used for tensile test and compression test and fractured samples during tensile test. Thirdly, type-A durometer was used to measure the hardness of the resultant composite as per ASTM 2240 standard.

SCANNING ELECTRON MICROSCOPE (SEM) EXAMINATION

SEM examinations on fractured and unfractured samples were done in Hitachi S-3400N model. Figure 3 shows the unfractured samples at different magnification. The bright spots on the surface represent the filler. It is clearly observed that at higher magnification fillers are uniformly distributed and minimal random aggregates are present. Also, they show no defects like voids or pullouts over the samples.

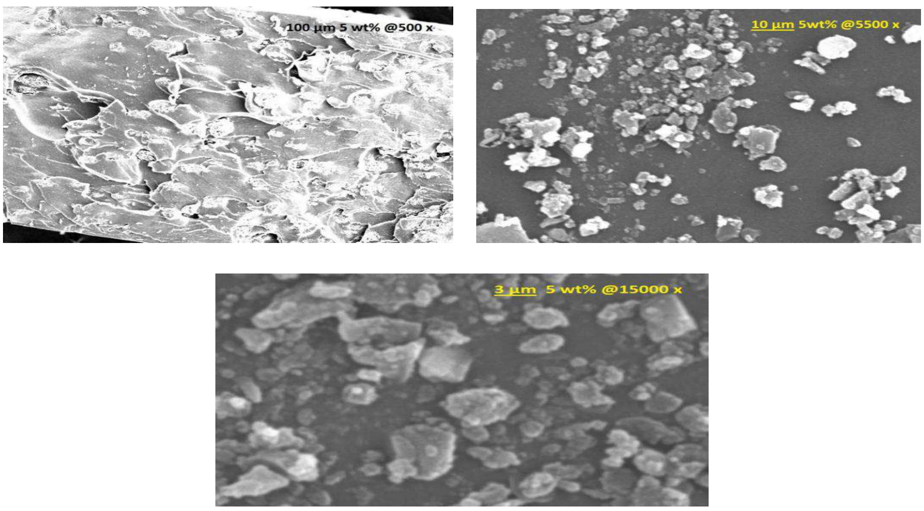

Figure 3: SEM images Observed from Unfractured 5 wt% MWCNT/Silicone Samples

Figure 4 shows SEM images of fractured samples with various magnification factors. The failure or crack initiation is visible in Figure 4(a) at 100x magnification. Figure 4 (b) represents crack development in the sample under tensile loading and transfer of load to MWCNT fiber from the silicone matrix under continuous stress. Figure 4 (c) & (d) shows a fractured surface with 5000 x and 15000 x magnification range where filler reinforcement embedded in the silicone matrix. The lagging of load transfer nature of matrix to reinforcement causes fracture or failure in the resultant composite samples.

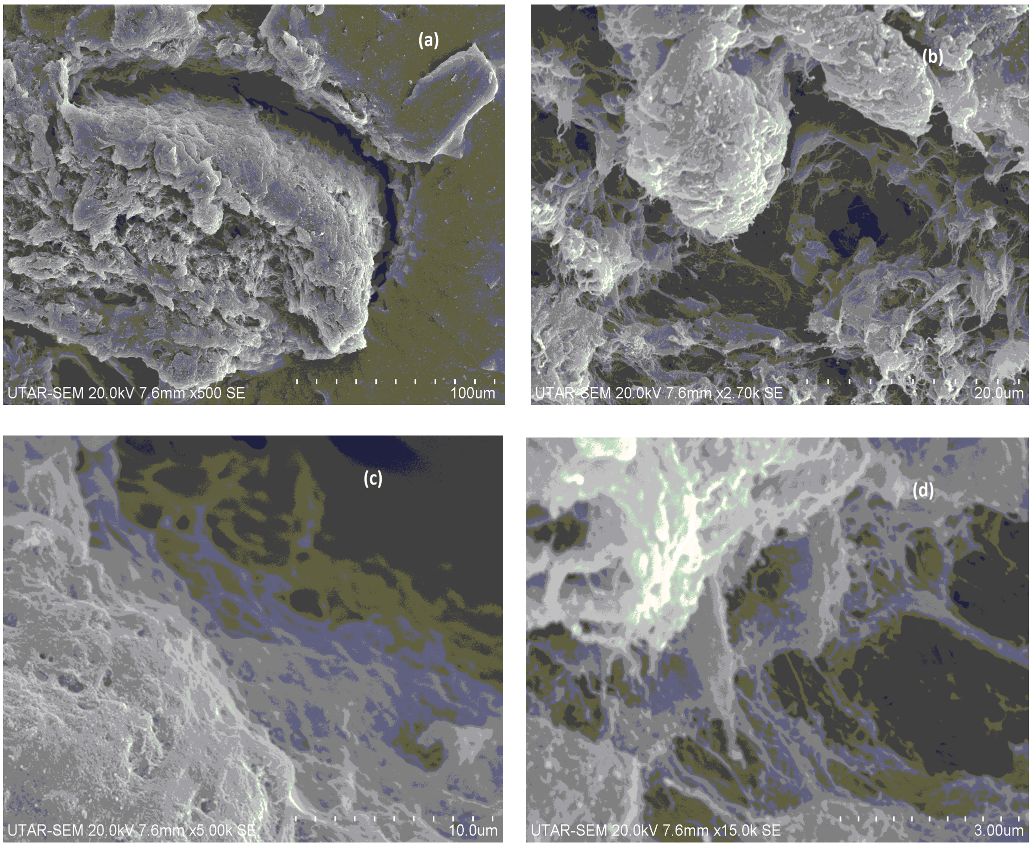

Figure 4: SEM Images of Fractured 5 wt% MWCNT/Silicone Samples

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR Analysis (Fourier Transform Infrared Spectroscopy) is a material analysis and characterization technique to identify and/or appraise surface contagion on the material, additives in a polymer etc. The chemical bonds or functional groups of the composite can be identified through the FTIR spectrum and the results can be used to enhance the quality control. FTIR examination on unfractured samples was conducted Nicolet iS10 FTIR spectrometer and characteristic of bonds in the chemical in a wavelength of light was absorbed. The FTIR absorbance and transmittance spectra of the sample is shown in Figure 5 (a) and 5 (b) respectively.

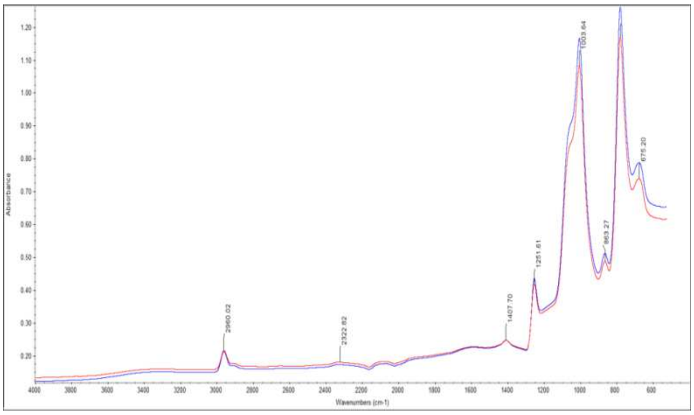

Figure 5 (a): FTIR Spectrum (Transmittance) of 5 wt% MWCNT / Silicone Composite

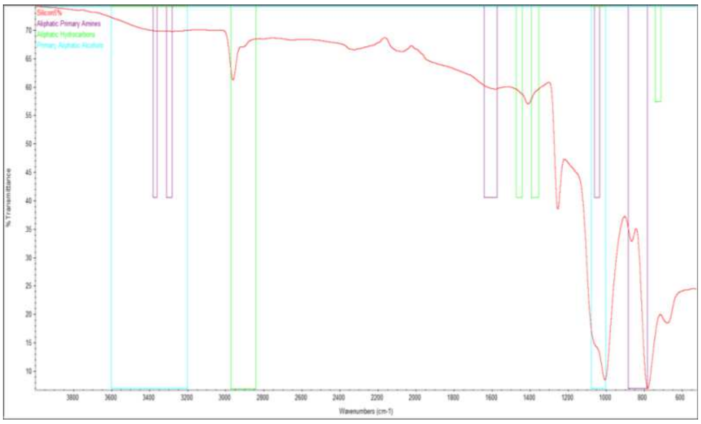

Figure 5 (b): FTIR Spectrum (Absorbance) of 5 wt% MWCNT/Silicone Composite

RESULTS AND DISCUSSIONS

The tensile strength, compression strength and hardness of unfilled silicone and MWCNT filled silicone composites are depicted in Figure 6-8. The result shows that inclusion of 5 wt% of MWCNT as filler material in silicone 40 elastomer has enhanced the mechanical strength and hardness substantially, but, slightly decreased the ductility of the material. The tensile strength of 5wt% filled MWCNT reinforced silicone composite is observed as 7.21 MPa which is 26% greater than unfilled composites. The corresponding tensile modulus of the composite is 1.673 MPa and strain at break is 707.07%. The elongation at break is observed as 235 mm. The result looks similar to the results reported in [6] in which authors attempted a MWCNT/NR composite. The process of synthesis is found good in the preparation of polymeric composite with uniform dispersion of fillers than other processes like melt blending or sonication methods. The compression strength of the sample is 18.6 MPa, which is 20% an increase. The hardness of the filled composite is found 64, which is 60% increment from the unfilled sample.

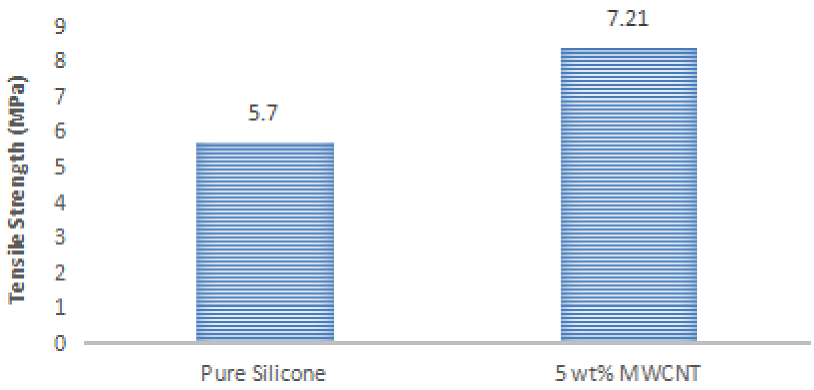

Figure 6: Tensile Strength of Unfilled and Filled Silicone Composite

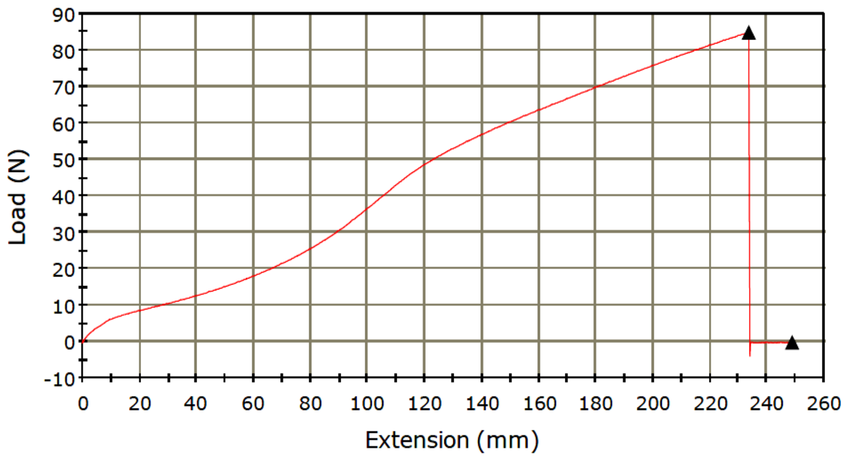

Figure 7: Elongation of 5% MWCNT Filled Silicone Composite

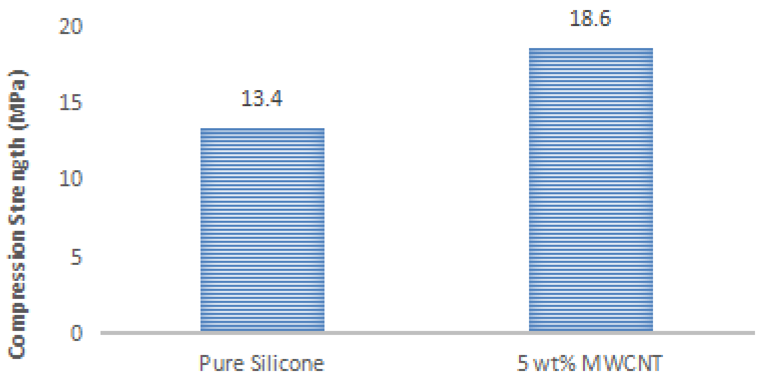

Figure 8: Compression Strength of Unfilled and Filled Silicone Composite

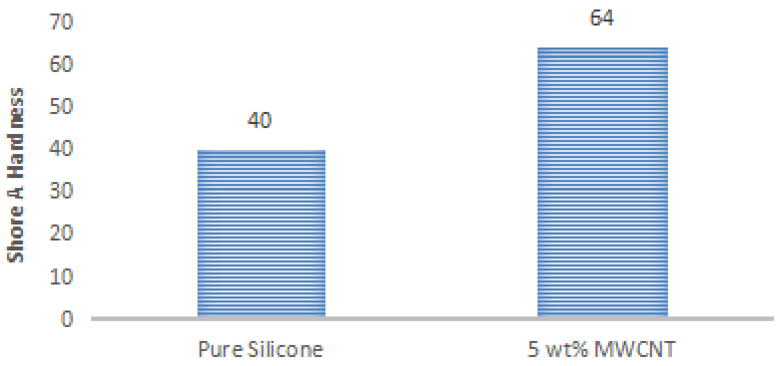

Figure 9: Hardness of Unfilled and Filled Silicone Composite

CONCLUSIONS

The aspiration of this research work is to examine the effect of MWCNT reinforcement on the mechanical properties of silicone 40 elastomer. Two roll mill with compression molding process was used for fabricating samples and tensile strength, modulus, elongation at break, strain at break, hardness and compression strength were measured. The tensile strength of the 5% MWCNT/silicone composite is 7.21 MPa which is 26% higher than pure silicone. Also, it is noted that the tensile modulus, strain at break, elongation at break of the MWCNT filled composite is 1.673 MPa, 707.07%, 235 mm respectively. It portrays MWCNT reinforcement considerably improved mechanical properties of silicone 40 elastomers. Compression strength and shore hardness also enhanced up to 20% (18.6 MPa) and 60% respectively. The targeted output with enhanced mechanical properties is preferably suitable for high strength aerospace applications like door mount, wing lateral panel, slat track, washers, rings, cargo systems and nose gear doors.

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